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SOLID-STATE DEVICE DEVELOPMENT FOR HIGH-PRESSURE
ENVIRONMENT

NAVAL SHIP RESEARCH AND DEVELOPMENT CENTER

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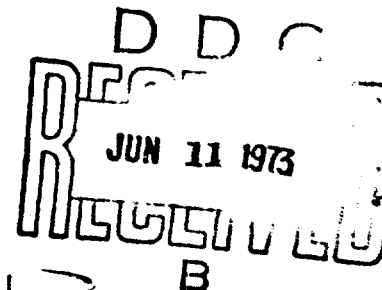
Bethesda, Md. 20034



SOLID-STATE DEVICE DEVELOPMENT FOR HIGH-PRESSURE ENVIRONMENT

By

M. J. Siegmann, V. W. Pugliese,
and D. E. Gilbert



PROPULSION AND AUXILIARY SYSTEMS DEPARTMENT

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Report 27-39

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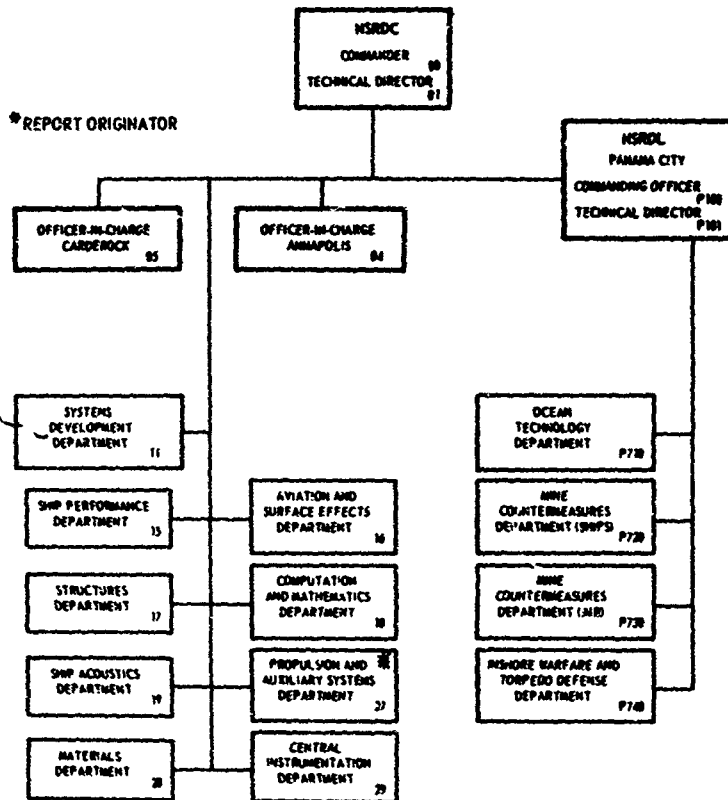
Solid-State Device Development for High-Pressure Environment

Report 27-39

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Naval Ship Research and Development Center
Bethesda, Md. 20034

MAJOR NSRDC ORGANIZATIONAL COMPONENTS



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- Hard-shell enclosures maintained at normal air pressure.
- Soft shell with a pressure compensating fluid.
- Free-flooded equipment.

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- Ocean environment on the soft shell.
- High hydrostatic pressure on the electrical and electronic components.
- Incompatibility of the components with the immersion fluid.
- Impulse-type pressures resulting from high acoustic energy and undersea blasts.
- Achieving positive buoyancy (shell, fluid and components).

Work directions necessary to alleviate anticipated and known problem areas are postulated and long-range goals established.

ADMINISTRATIVE INFORMATION

This work was accomplished under the Deep Ocean Electronics Program, Task Area XF 52545010, Work Unit P-612-123-A.

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INTRODUCTION

BACKGROUND

The need for electronics which will operate in a liquid environment that is pressure equalized to the ocean depths can be understood by a brief review of some of the considerations that stem from the Navy's interest in the deeper parts of the ocean. As submersibles are designed for greater depth, the ratio of hull weight to displacement increases rapidly and the sum of weights assigned to structures, propulsion, electrical plant and auxiliary systems rapidly approaches the total submerged displacement. As a result, for oceanographic submarines the payload is invariably a small percentage of the dry weight. The weight percentage for crew and instrumentation when averaged over several studies of oceanographic submersibles was 4%.¹ Specific vehicles follow this closely. For example, the ALVIN has a payload-to-dry-weight ratio of about 5% and the ALUMINAUT has a ratio of about 2%.²

As undersea exploration has continued, the demand for increasing payload has been well documented. A review³ of the validity of the original design considerations for the ALVIN, based on operating experience, reported that the operating specifications as written still appeared to be valid. However, less than a year's experience had forced recognition that the payload was not adequate and resulted in a requirement for a payload increase of 50% to be obtained by the addition of syntactic foam buoyancy. Nicholson and Cestone,⁴ in relating the deep submergence rescue vehicle (DSRV) electronics design and

¹Superscripts refer to similarly numbered entries in the Technical References at the end of this report.

development experience to future submersible needs, point out the following: (a) the heat generated by the electronics is difficult to remove due to limited hull wall space for heat exchangers, (b) the number of hull penetrators presents a safety problem, (c) interference reduction is essential and extremely difficult due to space and weight constraints, and (d) as much electronics as possible must be placed external to the pressure hull in order to alleviate the weight and space payload squeeze.

If the technology were available for design and development of circuits and equipments that would operate reliably when immersed in a pressurized liquid dielectric, most of the problems mentioned above could effectively be addressed. In addition to the deep submersible application, the technology would also have application to acoustic decoys, bottom installations, variable depth sonar, and submarines.

The objectives of this program are:

- The classification and characterization of materials components, technology, and system concepts as to their potential usefulness in ocean submergence application.
- The development of knowledge and technology to permit the utilization of as many electronic devices and components as practically possible to ensure that all types of system applications are possible.
- The development of a design and testing philosophy which can be used by designers to ensure long-term reliability of systems to be operated in the ocean environment.
- The documentation of the above information in a form useful to designers who are not familiar with the problem.

An underlying theme will be to document all results in such a manner that someone wishing to develop a system for use in the ocean environment should not have to do all the testing that was done on the original work. Classifying devices by manufacturer may not be of any value to a potential user since device fabrication techniques and materials may change at any time and frequently do.

APPROACH

The basic approach to the problem is to classify and separate the various observed effects into physical, physicochemical, short- and long-term reliability problem areas. It is expected that

intensive work in the various areas will permit successful synthesis and a more rapid, comprehensive solution to the overall problem.

More specifically, the approach is to:

- Develop a model of the electronic material, components, circuits and equipments immersed in a dielectric fluid:

- . That is consistent with reasonable overall constraints on the electronics such as environment, operating conditions, reliable performance, and cost.

- . That will recognize and accommodate the various physical and chemical interfaces between materials, components, circuits, dielectric fluid, and housing that require analysis and experimentation.

- . That will provide insight into the proper selection of dielectric fluid, housing, passivating, and potting compounds.

- Devise a scheme for presenting information on materials, devices, components, configurations, functions, failure modes, etc,* which will aid in the characterization of these parameters relative to pressure limits and fluid effects.

- Survey the field for existing devices, components, and circuit system fabrication techniques in order to identify representative classes for evaluation.

- Identify specific failure mechanisms and develop techniques to harden against such failure. Priorities for hardening-technique studies should be set according to universal application; i.e., try to identify a universal pressure-hardening technique and a universal fluid and contaminant-hardening technique and thoroughly investigate each.

- Devise test programs and implement them to determine long-term effects including pressure, fluid, contamination, temperature, and cycling.

- Fabricate representative and feasibility systems to demonstrate usefulness of the results.

*Abbreviations used in this text are from the GPO Style Manual, 1967, unless otherwise noted.

PROGRESS

LITERATURE SEARCH

At the start of the literature search, inquiries were made of the Library of Congress Reference and Referral Section, the Battelle Memorial Institute Information Research Center, and the National Oceanographic Instrumentation Center. The Library of Congress did a gratis search which was moderately helpful in that it confirmed our initial impressions. The bulk of the material retrieved was found through the iterative process of using the references provided by the reports and articles obtained.

The literature provides a reasonably good sampling of the passive discrete component area. In particular, the Scripps work is definitive. The reports on active components tend to be fragmentary and invariably focus on the question, "Will the component work in any application?". Unfortunately, the definition of what constitutes the application is usually missing. As a consequence, it has not been possible to do more than a generalization from the published data as to the nature of the physical or chemical failure mechanisms.

The state of the art with respect to component testing and the techniques available for examination of the component-fluid-equipment situation has been ascertained. The articles given as references in this report are representative of the search, but are by no means complete. In addition, the information obtained from the search determined in part the formulation of the approach.

BOUYANCY CONSIDERATIONS

Most submersibles designed for great depths are "weight critical;" that is, the weight of the hull, machinery, equipment and human payload is in excess of the weight of water displaced by the submersible. This occurs because of the large weight allocated to the pressure hull structure. Consider, for example, a welded spherical hull built to within feasible spherical tolerances and without stress relief. If the hull material is HY-150 steel, an operating depth of 20,000 feet results in a ratio of hull weight to displacement of unity. If the hull material is titanium 110, the hull weight-to-displacement ratio is unity for an operating depth of 30,000 feet. At 10,000 feet operating depth the weight-to-displacement ratio is about 0.5 for titanium 110 and approximately 0.65 for HY-150 steel.⁵

Weight-critical designs can attain neutral buoyancy by either enlarging the hull or by adding buoyancy materials outside the pressure hull. Both methods lead to increases in power needed for propulsion at design speed and battery capacity for the design endurance. Control devices also increase with craft size. All of these factors result in further increases in hull size.

Placing the electronics exterior to the pressure hull in the hydrostatic environment can do much to alleviate the payload squeeze caused by the weight of the hull. First, the material placed outside the hull displaces sea water and contributes to the buoyancy. Secondly, the space between the components can be filled with buoyant material. Therefore:

$$\text{Effective Density} = d_{el} \times \frac{v_{el}}{V} + d_f \left(1 - \frac{v_{el}}{V} \right),$$

where

d_{el} = average density of electronic material

d_f = density of the filler

v_{el} = displacement volume of the electronics

V = total volume.

Typical commercial electronics are packaged so that the packing factor, v_{el}/V is about 0.2. The average density of the electronic material is about 2.5 g/cc. If the dielectric fluid were a hydrocarbon of density 0.8 g/cc, the effective density of the overall package would be 1.14 g/cc. For depths less than 10,000 feet, one can attain positive buoyancy by a combination of hollow glass microspheres and the dielectric fluid. The microspheres have diameters in the range of 30 to 300 microns and density of about 0.33 g/cc.⁶ The packing factor for randomly packed uniform spheres is about 0.6 - 0.64. Microspheres with a distribution of diameters can be packed to a factor of 0.62 - 0.65 by slight mechanical vibration. Recalculating the effective density as above, for the use of electronics, microspheres and dielectric fluid, gives: effective density = $2.5 \times 0.2 + (0.35 \times 0.8 + 0.65 \times 0.33)(1 - 0.2) = 0.89$ g/cc.

Therefore, up to depths at which too many of the microspheres crush during screening, which is about 10,000 feet, this scheme would achieve positive buoyancy. In other words, a positively buoyant package can be created without the expense and increased structure size of the pressure shell to contain the electronics.

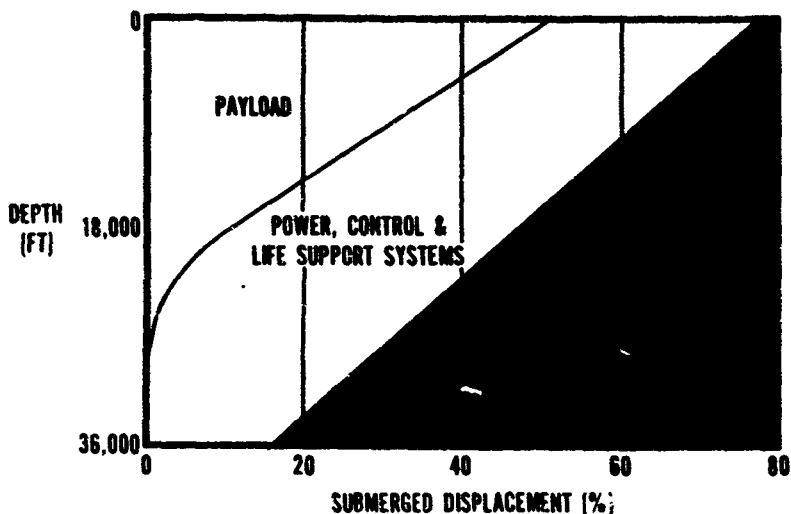
Below 10,000 feet, the ratio of weight to displacement of the hull gets much higher and the weight of the hull, machinery, equipment, and human payload is substantially in excess of 1 g/cc. Any reduction in buoyancy requirements makes the structure less weight critical. At depths below 10,000 feet, the use of syntactic foam and a liquid dielectric has promise. Syntactic foam is a solid structure in which the hollow glass microspheres are imbedded in a hardened resin. A typical density for the foam is 0.6 g/cc. Assuming that the packing factor for small, regularly shaped pieces of syntactic foam is 0.6, one can again calculate an effective density = $2.5 \times 0.2 + (0.4 \times 0.8 = 0.6 \times 0.6) 0.8 = 1.04$ g/cc. At any depth, this method can achieve positive buoyancy by a small decrease in the electronic material packing factor. For example, a packing factor of 0.15 instead of 0.2 would shift the effective density to 0.95 g/cc or positive buoyancy.

In summary, techniques have been outlined which can lead to the development of electronic packaging with positive buoyancy for all depths without the cost of pressure hulls and the added problem of a weight-critical design.

An interesting article analyzes the required buoyancy and the dry weight of the equipment penetrators and buoyancy material for two configurations of marine and special purpose equipment for a small submersible.⁸ The study was made because of the strong conviction that high power penetrators should not be allowed in pressure vessels designed for personnel occupancy. In one configuration, state-of-the-art electronics and normal partitioning inside and outside the hull was assumed. In the other configuration, the various equipments were partitioned so that only those portions necessary for display and control were inboard. A reasonable degree of multiplexing was assumed. The new wet-electronic configuration resulted in a decrease in required buoyancy from 1080 to 765 pounds and a total dry weight reduction from 5255 to 3615 pounds, or approximately a 30% reduction in each factor.

It is apparent that the existence of broadly based wet-electronics technology would provide the designers of Navy submersibles with many attractive alternatives for their assault

on the payload squeeze (see the figure below). In addition, an available wet-electronic technology will provide interesting options and alternatives for the developers of variable-depth sonars, acoustic decoys, and bottom or moored installations. The substantial efforts dedicated to the operation of electrical equipment and machinery exterior to the pressure hull in a liquid environment, requires a complementary effort for the control electronics.



The Payload Squeeze

DIELECTRIC FLUIDS

The desire to traverse the ocean depths has created requirements for dielectric fluids for which no precedent exists. The hostility of the environment and the sophistication of the equipment has placed new demands on fluids. The fluids will have to withstand exposure to ambient temperatures ranging from -2° to 150° C, cycling pressures to 14,000 psi, and a chemically corrosive environment. The fluid will be expected to shield solid-state components from the environment and prevent electronic failures due to energy transfer and contamination.

In order to select the best fluid, it was necessary to establish a scheme. First, to achieve positive buoyancy for the systems, only those fluids whose specific gravity was less than unity were considered. Second, the requirements of such a fluid, relative to the environment, were grouped into three categories, namely chemical, dielectric, and physical. The choice was then based on achieving superior qualifications in all three categories. No one fluid possessed the optimum properties in all three categories.

Fluids with Superior Chemical Properties

Table 1 was examined for a fluid that would be compatible with the electrical and electronic materials and also inhibit corrosion. In addition, the dielectric fluid should preferably not emulsify water and simultaneously retain a homogeneous fluid phase which would offer maximum dielectric properties.

For a first choice, MIL-S-21568A and VV-D-001078 were selected as exhibiting superior chemical properties. MIL-H-6083C was the second choice. The rationale for the selection is given below.

Compatibility

A fluid should be compatible with all the materials of the system to prevent accelerated corrosion and solution of materials commonly used in deep ocean technology. The tests⁹ were conducted by filling a vessel with a temperature transfer oil whose temperature was maintained at 60° C. A test cell, consisting of the materials which are utilized in deep ocean technology, was immersed in the dielectric fluid and placed in a temperature transfer oil. The pressure was maintained at atmospheric for 30 days. At the end of the test the specimens and the fluid were examined for changes in elongation, hardness, and tensile strength. Good signifies no change in the physical properties, fair indicates a slight change in physical properties (below 20%), and poor means a radical change in the physical properties.

Table 1
Dielectric Fluids, Chemical Properties

Base	Fluid	Compatibility at Atmospheric Pressure										Corrosion Protection			Viscosity after 1 hr	
		Butyl	Buna N	Buna S	Ethylene	Fluoro Sili- cone	Natural Rubber	Neoprene	Polyurethane	Sili- cone Teflon	Viton B	Aluminum			Viscosity after 1 hr	Water
												QD-250-48	QD-250-11	Copper		
Base	MIL-F-17111	Poor	Fair	Poor	Poor	Poor	Poor	Fair	NA	Poor	Good	Good	Good	Good	1	1
	MIL-H-5606B		Fair	Poor	Poor	Poor	Poor	Fair	NA	Poor		Good	Good	Good	1	1
	MIL-H-6081C		Fair	Poor	Poor	Poor	Poor	Fair	NA	Poor		Good	Good	Good	1	1
	MIL-H-81019B		Good	Poor	Poor	Fair	Poor	Fair	Good	Fair		Good	Good	Good	1	1
Petroleum	MIL-L-6081C		Good	Poor	Poor	Fair	Poor	Fair	Good	Fair		Poor	Fair	Good	1	1
	MIL-L-7870A		Good	Poor	Poor	Fair	Poor	Fair	Good	Fair		Good	Good	Good	1	1
	PR-1192		Good	Poor	Poor	Fair	Poor	Fair	Good	Fair		Good	Good	Fair	80	0
	Tellus 15		Good	Poor	Poor	Fair	Poor	Fair	Good	Fair		Good	Good	Good	12	12
Silicone	VV-I-530A		Good	Poor	Poor	Fair	Poor	Fair	Good	Fair		Poor	Good	Fair	1	1
	MIL-S-21568A		Fair	NA	NA	Poor	NA	Fair	Fair	Poor		Very	Very	Good	40	40
	VV-D-001078		Fair	NA	NA	Poor	NA	Fair	Fair	Poor		Very	Very	Good	40	40
	MIL-L-6085A		Fair	Poor	Poor	Good	Poor	Poor	Poor	Fair		Good	Good	Poor	72	1
Synthetic	MIL-L-7808G		Fair	Poor	Poor	Good	Poor	Poor	Poor	Fair		Good	Good	Good	72	0
	MIL-L-21699A	✓	Fair	✓	Poor	Good	Poor	Poor	Poor	Fair	✓	Good	Good	Good	40	1

NA-Not available.

Corrosion Protection

Fluids for deep ocean application must provide protection from the corrosive character of the environment and, in particular, sea water which has a high probability of entering the system. A fluid must display the ability to protect the system components from all forms of metal corrosion, that is, biological, cracking erosion, fretting, galvanic, general, intergranular, localized, selective, and stress.

The corrosion tests⁹ were conducted by immersing a metal specimen, 1 x 1 x 0.032 inch, in 270 ml of dielectric fluid at 60° C and atmospheric pressure for 30 days. The weight change of the metal specimen was determined and the following notation was adopted.

<u>Weight Change, mg</u>	<u>Designation</u>
0-50	Good
51-100	Fair
101-150	Poor
>150	Very Poor

Emulsion Stability

The need for a fluid which will emulsify water is a controversial subject. The proponents for a fluid with an emulsifying capability argue that emulsified water will wet the surface better than pure water due to the decrease in surface tension. This increased wetting will enhance heat transfer. They also propose that emulsified water would prevent short circuiting caused by water bridging an electrical gap. The opposition contends that emulsification is not necessary. If the specific density of water is greater than the dielectric fluid, then the water will settle to the bottom. A trap could be constructed such that mixing with the fluid would be eliminated. Our present position is that the emulsification capability is not necessary if the water trap functions properly.

The emulsion stability test procedure⁹ required 40 ml of dielectric to be stirred with 40 ml of distilled water for 5 minutes at 55° C. After standing for 1 hour, the volume of fluid, emulsion, and water were reported in table 1. A more sensitive emulsion stability test⁹ has been reported but data from the test has not been completed.

Fluids with Superior Dielectric Properties

A pressure-compensating fluid for electric and electronic components must have good dielectric properties and be inert to the action of electrical equipment. The dielectric quality of a fluid is determined by the dielectric breakdown voltage, dissipation factor, and electrical resistivity. The dielectric properties of a fluid result from its chemical composition and additives. Table 2 considers fluids with superior dielectric properties which are utilized in deep ocean technology. VV-D-001078 and Tellus 15 were the first and second choices for fluids with superior dielectric properties. The rationale for the selection follows.

Table 2
Dielectric Fluids, Dielectric Properties

Base	Fluid	Dielectric Breakdown Voltage v at 25° C		Dissipation Factor δ at 25° C		Resistivity, ohm cm at 25° C	
		As Received	Sea-Water Saturated	As Received	Sea-Water Saturated	As Received	Sea-Water Saturated
Petroleum	MIL-F-17111	23.2	NA	1.9	NA	8.2x10 ¹¹	NA
	MIL-H-5606B	27.8	7.2	2.0	2.1	3.0x10 ¹²	4.6x10 ¹²
	MIL-H-6083C	25.5	NA	6.1	NA	4.0x10 ¹⁰	NA
	MIL-H-81019B	22.4	NA	1.3	NA	2.6x10 ¹⁰	NA
	MIL-L-6081C	20.5	5	1.0	3.7	5.4x10 ¹²	9.0x10 ¹²
	MIL-L-7870A	30.4	NA	14.4	NA	7.6x10 ⁹	NA
	PR 1192	21.6	NA	20	NA	7.4x10 ⁹	NA
	Tellus 15	30	0.8	NA	NA	8.0x10 ¹³	NA
	VV-I-530A	25	8.9	2.0	2.3	5.2x10 ¹³	15x10 ¹³
Silicone	MIL-S-21568A	26.2	16.4	2.0	1.5	2.2x10 ¹³	2.4x10 ¹³
	VV-D-001078	26.3	NA	0.00004	NA	1.0x10 ¹⁴	NA
Synthetic	MIL-L-6085A	26.9	NA	60	NA	8.0x10 ⁸	NA
	MIL-L-7808G	25.6	NA	5.5	NA	2.2x10 ¹⁰	NA
	MIL-L-23699A	NA	NA	NA	NA	NA	NA

NA-Not available.

Dielectric Breakdown Voltage

During usage, dielectric properties are affected by arcing and contamination. Arc discharge through the fluid can produce carbon, silica, and chemical changes in the dielectric which could reduce its breakdown voltage and electrical resistivity to unacceptable limits. Likewise, contamination of the fluid by impurities or sea water can reduce the breakdown voltage and resistivity to unacceptable limits. For optimum fluid protection against arcing, a high dielectric breakdown voltage is desired. A tentative measure of acceptable dielectric breakdown voltage for dielectric fluids has been set at 15.0 kv (minimum) at a 0.05-inch electrode gap. The dielectric breakdown voltages were measured according to ASTM procedure D-877 with the exception of a 0.05-inch electrode gap.⁹

Dissipation Factor

The requirements that fluids inhibit corrosion and lubricate dictate the utilization of polar and water-soluble additives. These polar materials not only lower the dielectric breakdown voltage and resistivity, but also increase the dissipation of electrical energy as heat. The ideal dielectric fluid would have a dissipation factor of 0%. A relative measure of acceptable dissipation factor for dielectric fluids has been fixed at 2.0%. The dissipation factors⁹ were determined from the measurement of the loss angle of a fluid-filled cell on a capacitance bridge.

Resistivity

Maximum protection against failures of electrical and electronic components dictates that the dielectric fluid exhibit a minimum resistivity of 3.0×10^{11} ohm-cm, which is the tentative measure of acceptable resistivity.

Fluids with Superior Physical Properties

Optimum physical properties for the fluid require that it have a specific gravity of less than unity, high flash point, low isothermal compressibility, and low viscosity. Table 3 was formulated for fluids with a high probability of meeting the requirements set forth above. Examination of table 3 resulted in the selection of MIL-S-21568A and Tellus 15 as the first and second choices. The rationale for the selection is indicated below.

Table 3
Dielectric Fluids, Physical Properties

Base	Fluid	Density, g/cc-l				Flash Point	Isothermal Compressibility		Viscosity, cs			
		at 15,000 psi					at 15,000 psi		at 15,000 psi			
		15,000	15,000	15,000	15,000		15,000	15,000	2° C	38° C	2° C	38° C
Petroleum	MIL-E-17111	NA	NA	NA	NA	104	NA	NA	75	NA	28.8	NA
	MIL-H-56069	0.866	0.908	0.840	0.887	102	0.308	0.353	41	27	14	58
	MIL-H-6083C	0.870	0.912	0.845	0.892	110	0.307	0.351	10	174	12	43
	MIL-H-81019B	NA	NA	NA	NA	100	NA	NA	17	NA	7.2	NA
	MIL-L-6081C	0.881	0.920	0.857	0.900	152	0.283	0.318	54	229	10	70
	MIL-L-7870A	NA	NA	NA	NA	141	NA	NA	71	NA	10.3	NA
	PR-1192	NA	NA	NA	NA	191	NA	NA	300	NA	41.9	NA
	Tellus 1	0.883	0.921	0.860	0.910	149	0.275	0.366	60	NA	10.8	NA
Silicone	VV-1-530A	0.895	0.932	0.870	0.912	163	0.265	0.307	57	260	9.7	77
	MIL-S-21568A	0.848	0.901	0.801	0.860	46	0.466	0.522	1.19	4.3	0.76	3
	VV-D-001078	0.957	1.09	0.924	1.02	179	0.813	0.627	17.9	44	8.66	35.5
Synthetic	MIL-L-6085A	NA	NA	NA	NA	196	NA	NA	55	NA	12.7	NA
	MIL-L-7808G	NA	NA	NA	NA	213	NA	NA	75	NA	17.3	NA
	MIL-L-23699A	NA	NA	NA	NA	254	NA	NA	160	NA	25.7	NA

cs-Centistokes.

NA-Not available.

Density

Positive buoyancy is a desirable feature in a submersible. The utilization of a fluid whose density will remain less than 1 g/cc within the pressure and temperature range will aid in achieving positive buoyancy.

The change in density with temperature and pressure must be given careful consideration since the pressure effect is opposite to that of temperature. Density increases with pressure and decreases with temperature. The dependence of density on temperature and pressure is seen on examining the temperature dependence of the isothermal compressibility.

The density data were calculated mathematically from the isothermal compressibilities.

Flash Point

Extreme care must be taken in handling fluids with flash points below 150° C. The flash points recorded in table 3 were obtained according to ASTM D-943 procedures.

Isothermal Compressibility

Deep submergence vehicles are plagued with high weight-to-volume ratio. One reasonable solution to alleviate this problem would be to pressure compensate parts of the system. Pressure compensating the system would reduce the thickness of the exterior shell, but it would demand that the fluid be isothermally incompressible. For if the fluid did collapse, the system's shell would implode.

The isothermal compressibilities of several fluids, measured from the volume decrease under applied pressure and constant temperature,¹⁰ are recorded in table 3. A comparison of these values with the anomalous compressibilities of water reveals that the dielectrics are less compressible.

Isothermal Compressibility of Water

at 2° C : $0.35 \text{ psi}^{-1} \times 10^5$

at 38° C : $0.30 \text{ psi}^{-1} \times 10^5$

Viscosity

Viscosity is one of the most important single properties of a fluid because convection, diffusion, and thermal conductivity depend on viscosity.

Ideally, it is desired that the viscosity remain constant over the whole temperature and pressure range so that heat and mass transfer through the fluid to the surface of solid-state components can be predicted. Viscosity increases with pressure and decreases with temperature.

The attributes of lower viscosity fluids include: inhibition of formation of solid products, "clinkers," between contact surfaces; increased heat dissipation; and enhanced emulsification of water, thus minimizing the reduction of the dielectric breakdown voltage and insulation resistance.

The kinematic viscosities, measured according to acceptable techniques⁹ and ASTM-D445, are recorded in table 3.

Dielectric Fluid Selection

After careful examination of the chemical, dielectric, and physical properties it can be concluded that the silicones are generally superior. MIL-S-21568A is our choice for the silicone fluid. However, the susceptibility of silicone-base fluids to chemical degradation by acids and bases and the ease with which they dissolve solid silicones (e.g., silicone molding compounds) warrants the choice of an alternate fluid which possesses different chemical properties. Tellus 15 was chosen as the alternate fluid, but based on fragmentary information Tellus 11 appears competitive. However, solidification of Tellus 11 at 5,000 psig and 2° C precludes its use below 10,000 feet.

HOUSING MATERIALS FOR FLUIDS AND DEVICES

Requirements for the housing material include high strength-to-weight ratio, good thermal conductivity, inertness to both sea water and dielectric fluid and the capability to be machined and formed. Tables 4 and 5 consider various metals, alloys, and synthetics. Where numerical values are not given, relative judgements are made and designated as poor, fair, good, or excellent.

Table 4 - Housing Materials, Physical and Mechanical Properties

Prop	Material	Typical Physical Constants		Typical Mechanical Properties		
		Specific Gravity	Conductivity, 12"-212° F Btu/ft ² /hr/° F/in	Modulus of Elasticity 10 ⁶ psi	Tensile Strength 10 ³ psi	Workability
Metals and Alloys						
Aluminum	5000 Series	2.69	80	10.2	38	Good
	6061	2.72	115	10	27	
Copper	Cu-Ni (70-30)	8.97	200	26	70	Good
	Cu-Ni (90-10)	8.97	310	18	50	
	Ni-Al-Bronze	7.80	490	17	91	
	Ni-Al-Mn-Bronze	8.36	700	15	90	
Lead	60% Pb-40% Sn	8.91	330	2.0	6.8	Good
	Hastelloy C	8.88	61	26	130	Poor
Nickel	Inconel 625	8.52	100	31	80	Fair
	Inconel 718	8.61	86	31	190	
	Monel 400	8.88	174	26	95	
	Ni-CR-Mo Alloy	8.19	61	24.5	75	
Stainless Steel	Alloy 20	7.77	103	28	100	Good
	Type 316	7.97	113	28	125	
Titanium	Pure	3.41	118	14.9	67	Good
	Ti-6Al-4V	4.44	52	16.5	130	
	Ti-6Al-2CL-1Ta/ 0.8 Mo	4.41	59	17.7	120	
Other						
Glass	Pyrax	2.33	7.5	9.5	10	Fair
	Polyethylene type 1, low density	0.92	2.3	0.02	2.0	Good
Plastics	Polystyrene					
	general purpose	1.05	0.9	0.45	7.0	Fair
Elastomer	Synthetic Foam	0.65	0.7	0.4	4.0	Good
	Neoprene	1.25	1.4	0.0015	1.0	Good
	Silicone	1.04	1.2	0.0009	0.8	

Table 5
Housing Materials, Chemical Properties
and Estimated Overall Performance

		Typical Chemical Properties				
Class	Material	Pitting Rate, sea-water velocity 3 ft/sec, mil/hr	General Sea-Water Resistance	Biofouling Resistance Velocity 3 ft/sec	Coatability	Estimated Performance
Metals and Alloys						
Aluminum	6000 Series	20	Good	Poor	Fair	Poor
	6061	80	Fair	Poor	Fair	Poor
	7075-T6 (90-10)	1-5	Good	Fair	Good	Good
	7075-T6 (10-90)	1-5	Excellent	Good	Unnecessary	Good
	Ni-Al-bronze	2-9	Good	Fair	Good	Fair
	Ni-Al-Mo-bronze	1-2	Good	Fair	Good	Fair
Steel	CG PL-40 Ss	Negligible	Excellent	Poor	Fair	Fair
Inconel	Hastelloy C	Negligible	Excellent	Poor	Good	Fair
	Inconel 625	Negligible	Excellent	Poor	Good	Fair
	Inconel 718	Poor	Good	Poor	Good	Fair
	Model 400	5-15	Poor	Fair	Good	Poor
	Ni-Cr-Mo Alloy	Negligible	Good	Poor	Good	Good
	Alloy 20	5-10	Good	Poor	Fair	Poor
Stainless Steel	Type 316	50	Poor	Poor	Fair	Poor
Titanium	Pure	Negligible	Excellent	Poor	Good	Good
	Ti-6Al-4V	Negligible	Excellent	Poor	Good	Good
	Ti-6Al-2Cu-1Zr-0.8Mo	Negligible	Excellent	Poor	Good	Excellent
	ITA/0.8Mo					
Other						
Glass	Pyrex	N/A	Excellent	Good	Poor	Poor
Plastic	Polyethylene	N/A	Insufficient Data	Poor	Poor	Poor
	Type 1, low Density					
	Polystyrene general purpose	N/A	Insufficient Data	Poor	Poor	Poor
	Synthetic foam	N/A	Excellent	Poor	Excellent	Good
Rubber	Neoprene	N/A	Good	Poor	Poor	Poor
	Silicone	N/A	Good	Poor	Poor	Poor
N/A - Not applicable.						

N/A - Not applicable.

Of the materials considered, a new titanium alloy (Ti-6Al-2Cu-1Zr-0.8Mo) is the first choice. Characteristics of this titanium alloy include high strength-to-weight ratio (helping to achieve positive buoyancy); good thermal conductivity; machinability; weldability; high strength without heat treating after casting or welding; and resistance to pitting, corrosion, and biofouling in seawater when coated. A major disadvantage is its lack of availability. Therefore, other materials (e.g., aluminum) are being considered.

Future efforts will analyze the selected housing material to determine compatibility with the dielectric fluids, capability for bonding dissimilar metals, and the resultant effects due to heat and pressure, in addition to design requirements and techniques to be utilized to fabricate the first test vessel.

References 11 , and 13 may be consulted for additional information on the effects of seawater on metals.

DEVICE CLASSIFICATION

Table 6 is included in this report to provide a general understanding of the short-term effects of high hydrostatic pressure cycling (0-14,000 psi) on electrical and electronic components.

Resistors exhibit a decrease in resistance under pressure. Carbon types, which are the most pressure sensitive, vary 20% or more, while the metal-film and wire-wound resistors vary less than 10%.

Capacitors, depending on the type and size, pose a similar problem but with an increase instead of a decrease in value. The dry-type capacitors, namely glass and ceramic, only suffer slight changes with pressure (less than 5%). The wet capacitors, tantalum, paper, and especially electrolytic, are characterized by a considerable increase in capacitance and subsequent case collapse and functional breakdown.

Inductors display the greatest sensitivity to high pressure. Iron-core types are characterized by large and often erratic variations with some permanent change in inductances and Q. Air-core inductors are less sensitive to pressure. With either type, maximum change occurs during pressure cycling and appears to stabilize at a lower value after soaking.

The performance of semiconductor devices under pressure vary widely depending upon size, fabrication techniques, materials, and packaging. Most short-term failures are caused by case deformation or collapse resulting in open circuits and in some instances short circuits. For example, semiconductor components housed in metal cans, such as the T0-3 and T0-5 cases, were crushed. Many of the same type could be operated by puncturing the case prior to pressurization so that oil could enter the housing. Other cases made of epoxy and ceramic cracked under pressure. Characteristics of diodes, SCR's, and transistors, which did not experience catastrophic failure, varied from less than 10% to as much as 50%.

Mechanical devices such as servos, solenoids, and contactors are not considered to be reliable for deep ocean use. The functions of these devices may be replaced with solid-state circuit techniques. In some cases, pressure hardening of the mechanical devices may prove satisfactory.

Table 6
Pressure Effects on Electronic Devices

	Limits of Values Tested	Type Tested	Change in Value	Failure Mode	Estimated Performance
RESISTORS					
Carbon composition	<1 m Ω , <2 watts	-	Negative, >20% to failure	Case deformation	Poor
Deposited carbon	<100 k Ω , <1/2 watt	-	Negative, >10%	N/A	Fair
Metal film	<1 m Ω , <1 watt	-	<5%	N/A	Good
Wire wound	<100 k Ω , <10 watts	-	Negative, <10%	N/A	Good
CAPACITORS					
Dry					
Glass	<0.001 pf, 500 vdc	-	<2%	N/A	Good
Ceramic	<0.5 pf, <10 vdc	-	<5%	N/A	Good
Wet					
Tantalum	<100 μ f, <45 vdc	-	Positive, >5% to failure	Case deformation	Poor
Paper	<8 μ f, <500 vdc	-	Positive, >10% to failure	Case deformation	Poor
Electrolytic	<85 μ f, <50 vdc	-	Positive, >15% to failure	Case deformation	Poor
INDUCTORS					
Coil					
Air core	<100 h	-	Negative, <10%	N/A	Good
Iron core	<2 μ h	-	>10%	N/A	Fair
Transformer	Low power	-	10%-50%	N/A	Poor
SOLID-STATE COMPONENTS					
Diode and SCR					
Glass	-	Low power	<10%	N/A	Good
Epoxy	-	Low power	>10% to failure	Case deformation	Poor
Metal	-	High power	>10% to failure	Case deformation	Poor
Transistor					
Epoxy	-	Low power	>10% to failure	Case deformation	Poor
Metal	-	High power	Failure	Case deformation	Poor
IC, MSI, LSI, MOS, thin and thick film devices	-	-	-	-	Unknown (probably poor)
SERVO	-	-	-	-	Unknown (probably poor)
SOLENOID	-	-	-	-	Unknown
CONTACTOR	-	100 amperes 120 volts	N/A	Clickers	Poor

Note: Failure = Crack, break, or change in excess of 50%.

m Ω - megohms.
k Ω - kilohms.
pf - picofarads.
h - microhenries.
N/A - Not applicable.

SCR - Silicon controlled rectifier.
IC - Integrated circuit.
MSI - Medium-scale integration.
LSI - Large-scale integration.
MOS - Metal-oxide-silicon.

The information shown in table 6 and discussed above is a summary of data found in references 14 through 18. Much of the information, knowledge, and results of such studies are of little value to potential users because the reported results are very incomplete. The information was probably useful to the investigator in some particular system he was building, but the lack of complete or even adequate characterization of devices tested makes it unreasonable to interpret the results for other applications. A scheme has been developed, for the present program, to avoid this problem and to allow for identification of acceptable devices as well as problem areas. The scheme has been made flexible to accommodate the dynamic nature of device technology, wherein new devices, and improvements in old devices such as reliability and reduced cost and size, are consistently being pursued.

Table 7 is representative of the scheme to be utilized. Seven separate sorts of pertinent facts for each device evaluated are shown. Manufacturers information relative to geometry, fabrication techniques, materials, and packaging will be combined with test data to clearly define the capability of the device and permit classification. Innovations such as pressure and fluid hardening techniques will be employed, tested, and documented as indicated.

Incorporation of data in the format established in table 9 will provide:

- A potential designer with the kind of information necessary to develop a system for use in the ocean environment without resorting to individual component testing.
- The capability to judge the operation of untested devices in which a manufacturer's fabrication method changed.
- The means to update information on new devices in which there is no precedent.
- A method to reduce the time necessary to achieve meaningful patterns and decrease the quantity of devices to be evaluated within a given class.
- An understanding of the limitations of particular devices, in terms of maximum pressure, soaking time, number of cycles, and functions.

Table 7
Device Classification Scheme (Headings of Seven Separate Tables)

Housing Type and Material											
Designation	Mfr and Date	Func- tion	Element Geometry	Housing Type	Housing Material	Element Bond	Lead Type and Bond	Pass- ivity	Pressure & Cycle Limit	Pressure Hardening Effects	Fluid Hardening Source
Element Geometry											
Designation	Mfr and Date	Function	Housing Geometry	Housing Type	Housing Material	Element Bond	Lead Type and Bond	Pass- ivity	Pressure & Cycle Limit	Pressure Hardening Effects	Fluid Hardening Source
Element-Substrate Bond											
Designation	Mfr and Date	Function	Element Geometry	Housing Type	Housing Material	Housing Element Bond	Lead Type and Bond	Pass- ivity	Pressure & Cycle Limit	Pressure Hardening Effects	Fluid Hardening Source
Passivation											
Designation	Mfr and Date	Function	Element Geometry	Housing Type	Housing Material	Housing Element Bond	Lead Type and Bond	Pass- ivity	Pressure & Cycle Limit	Pressure Hardening Effects	Fluid Hardening Source
Pressure Limit											
Designation	Mfr and Date	Function	Element Geometry	Housing Type	Housing Material	Housing Element Bond	Lead Type and Bond	Pass- ivity	Pressure & Cycle Limit	Pressure Hardening Effects	Fluid Hardening Source
Fluid Effects											
Designation	Mfr and Date	Function	Element Geometry	Housing Type	Housing Material	Housing Element Bond	Lead Type and Bond	Pass- ivity	Pressure & Cycle Limit	Pressure Hardening Effects	Fluid Hardening Source
Function											
Designation	Mfr and Date	Element Geometry	Housing Type	Housing Material	Element Bond	Lead Type and Bond	Pass- ivity	Pressure & Cycle Limit	Pressure Hardening Effects	Fluid Hardening Source	Source
Mfr - Manufacturer.											

Mfr - Manufacturer.

The need for consideration of device limitations relative to pressure and cycles was made evident after analysis of data compiled at the Hawaii Laboratory of the Naval Undersea Research and Development Center and at this activity. References 19 and 20 discuss devices pressure tested in Tellus 11 oil at the Hawaii Laboratory. Reference 19 indicates 100% survivability for transistors utilizing the Fairchild Micro Pac and the Motorola T packages after 200 cycles (0-15,000 psi). Reference 20 indicates approximately 98% survivability for 742 samples of the Signetics type N5741A operational amplifier when cycled 100 times (0-5,000 psi). The housing of the survived devices in reference 19 was Dow Corning 308 and in reference 20 was Dow Corning 306. These are silicone-plastic molding compounds designed for encapsulation of semiconductor devices by transfer molding. Theoretically, the advantages of this type of molding compound include, absence of water or other contaminant by-products released during curing, ease and extent of crosslinking, wide temperature range over which they are stable, and their excellent electrical properties over an extreme range of environmental conditions. However, data compiled at this activity²¹ on 15 samples of the General Electric power transistors of the D44R4 and D43C8 types resulted in 70% survivability after 1000 cycles (0-13,500 psi). The housing of General Electric transistors is Dow Corning 306, the same as that used for the Signetics type N5741A operational amplifiers. Further, attempts made at this activity to ascertain the required number of cycles needed to assure reliable operation of various plastic encapsulated devices have proved futile. Plots of failure versus pressure cycles did not produce a meaningful pattern. It is, therefore, not clear for a given pressure level how long a device will last past a given number of cycles, whether one can assure reliable operation for a specific number of cycles or the number of testing cycles required to ensure reliable operation thereafter and for how long.

Table 7 indicates that device function will be a major sort. This can be easily understood if one considers high impedance circuitry such as field-effect transistor switches. A minute amount of moisture seepage reducing the insulation resistance below 10^{10} ohms would cause a malfunction. This same amount of seepage would be tolerable in lower impedance circuits. It is apparent that some devices are inherently more difficult to protect against the electrochemical effects of dielectric fluid contaminants than others. The more sensitive devices will be subjected to coating or encapsulation and study.

IDENTIFICATION OF DEVICE FAILURE MODES

Failure modes can be classified into three general categories: (1) hydrostatic, (2) fluid interaction, and (3) acoustic noise and shock. The hydrostatic failures can be further classified into those which result from geometry, dimensions, material strength, and incompatible materials. These failures can show up in any number of ways, but a pattern often exists which permits their identification and prevention. For example, voids are the most prevalent cause of hydrostatic failure. This failure mode can be assessed for devices by considering the geometry, dimensions, and material properties, then choosing a technique for preventing collapse. Two techniques available for hardening are potting with a structurally supportive material or flooding. Another hydrostatic failure mode is the electrical shorting of a device due to the deformation of some part of the device, for example, shorting between the contact posts in a transistor. Other failure modes are stressing of materials beyond their elastic limits to produce a fracture and bonding of two materials with different compressibilities to produce stress.

The second class of failure modes results from the fluid environment which is the transport medium for contaminants. These may be caused by the fracture of some encapsulation material, deliberate flooding, or simply the lack of the use of any passivation scheme for device elements. These problems are not easy to classify; however, there are at least three categories: (1) filling of voids, (2) electronic interaction, and (3) chemical interaction. The filling of voids by the immersion fluid can cause the device to explode when pressure is suddenly released. The second category involving electronic interaction is as broad as the types of elements which will be used. For example, the fluid or a contaminant transported by the fluid may collect on the surface of an insulating or semi-insulating element and influence electronic charge distribution in the element. Thin film resistors would be very susceptible to this failure mode. Another example is the diffusion of ions, such as sodium, into critical elements or device parts resulting in degradation of the device functions, e.g., a shift in MOS transistor gate voltage with any sodium ions which could find their way into the oxide or the oxide silicon interface. The bonding of the passivated silicon chip to a gold-plated header presents a pathway for moisture and ionic contaminants along the passivant-gold interface since the adherence of plastics to gold, kovar, nickel alloys, solder, solder plating, and cadmium is poor.

Chemical interaction of the fluid or fluid contaminants is a serious problem. For example, silicone fluid softens the silicone passivant on a device. Ionic contaminants can come from

many sources. In one instance,²² the degradation of a curing agent in a rigid phenolic outer case of a microdiode produced ammonia which permeated a silicone barrier coating between the phenolic and the semiconductor surface. The ammonia caused an increase in the leakage current of the diode. Chloride ions, from a source like sea water, corrode aluminum leads which result in an open circuit or a high resistance. The chloride ions can also come from hydrochloric acid found in the package. Water vapor reacts with aluminum in the presence of chloride ions to form aluminum hydroxide and regenerates the chloride ion in the form of hydrochloric acid for further attack on the aluminum.

The third type of failure mode is acoustic noise and shock. The electronic equipments which will operate immersed in a liquid will be expected to function in conjunction with a vehicle or stationary platform of some type that will be engaged in a military action. As a consequence, the underwater tactics will involve the generation of acoustic noise that will impinge on the electronics. The noise comes from a variety of generators such as hull, propeller, machinery, sonar, etc. In addition, the presence of shock waves of various amplitudes is a combat reality. A brief examination will be made of the possible consequences of exposing the electronic components and equipments to this acoustic environment.

The velocity of sound is approximately 1500 m/sec near the surface and increases by about 8% at a depth of 7000 meters. For our consideration, dispersion, temperature, and salinity effects are unimportant in terms of sound velocity. Sound attenuates exponentially as a function of frequency; at 100 Hz the attenuation is approximately 0.0001 db/kiloyard, while at 10 kHz the attenuation is nearly 1 db/kiloyard.

In general, solid-state electronics are not subjected to microphonics when operated in air, even in high sound ambients. By way of comparison, a subway train passing through a station (a high acoustic ambient in air) generates an acoustic sound level of 10^{-6} watts/octave bandwidth.²³ This power level in sea water develops an rms pressure amplitude of about 400 dynes/cm². This value is 126 db above the acoustic reference of 0.0002 dynes and would constitute a substantial ambient under most circumstances.

The normalized acoustic impedance of an object immersed in a propagating medium is a function of D/λ , where D is approximately the dimension of the object and λ is the acoustic wavelength. The normalized acoustic impedance of the object is unity

for $D/\lambda \gg 1$, and is approximately $1/\lambda^2$ for $D/\lambda \ll 1$. The wavelength, λ , for 100 Hz in water is 15 meters and the λ for 10 kHz is 15 centimeters. These wavelengths are large compared to that of the electronic components and, therefore, the coupling of any available acoustic energy to the components would be small because of the acoustic mismatch. In fact, it is only when the wavelength is smaller than the size of the chassis and equipment that the impedance becomes matched for the higher frequencies. The level of ambient acoustic powers expected is easily absorbed by the chassis or equipment.

Since the device size is small compared to the wavelengths, the device is, in effect, experiencing a constant pressure over its surface which varies up and down. Essentially, there is no flexure due to variable stress over the surface. In summary, the acoustic noise ambient appears to be rather benign.

Shock waves present a somewhat different problem. If one assumes that the shock wave front has a rise time of about 0.1 msec and a substantial amplitude, say 3,000 psi, a problem may exist. Assuming a linear rise in pressure amplitude and an electronic object of 1-cm length, a pressure difference of 200 psi would appear along the length. This pressure differential is inconsequential compared to that to which semiconductors used as pressure transducers are subjected. Therefore, the bending moments caused by the pressure differential of the shock wave will not generally present a problem to the components. However, possible effects on the overall enclosure will be considered at a later date.

INFLUENCE OF PRESSURE ON DEVICES

Mechanical stress or strain changes the lattice spacings in materials and also destroys the crystal symmetry. It is often this symmetry change that induces the more pronounced changes in electrical properties. When the lattice spacings and symmetries are altered, they, in general, cause changes in almost all the material parameters which contribute to the electrical properties. In addition to purely dimensional changes, one finds changes in such important parameters as the amplitude of vibration of the atoms about their normal lattice sites, the energy band structures, the inter- and intra-atomic interaction, and the impurity-lattice interaction. This list is not complete but points out the magnitude of the problem.

Changes in lattice vibrations in metals, for example, cause changes in the scattering cross sections; hence, the electronic mobility changes. With changes in energy bands come changes in

the Fermi energy which, in turn, can cause drastic changes in charge-carrier populations. The latter is particularly true for alkali metals and semiconductors. In semiconductors, slight changes in band structure can cause orders of magnitude changes in minority carrier densities. Many of these effects are not well understood. Any comprehensive study of pressure effects on contemporary electronic properties will, of necessity, require a dynamic test program to test those materials and properties for which there is no information available. For example, fundamental information on the dielectric properties of insulators under stress is virtually nonexistent.

Consider the effects of stress on resistors. This is one of the easier devices to characterize due to its simplicity of material, phenomena, and configuration. Here the primary concern is with the electrical conductivity (piezoresistance and elastoresistance). Fortunately, there has been much work in this field.²⁴ Also fortunately, hydrostatic pressure has little effect on the crystals and homogeneous materials used.

Practically no work has been done on the experimental characterization of dielectric materials under pressure. Also, there is little theoretical information on pressure effects, dielectric constants, loss tangents, etc. There has been some work on ferroelectrics such as BaTiO_3 ,²⁵ and SrTiO_3 ,²⁶ in which large changes in the dielectric constants have been observed with pressure. The dielectric constant of these materials decreases with pressure while the loss tangent first increases and then decreases as pressure is increased. Some of the single cubic crystals of the alkali halides have also been investigated.²⁷ The optical properties of materials are closely related to the dielectric properties, and some insight has been gained into the dielectric properties of materials by studying their optical properties under pressure.

Inductors and other magnetic devices are susceptible to the magnetostrictive effect. Research on this phenomenon dates back to 1898,²⁸ with much study on iron and nickel. The famous curve of Bethe and Slater, which relates the exchange coefficient to nearest neighbor distance, gives one an immediate appreciation of the expected stress behavior of magnetic properties. In fact, stress has often been used as a tool to study magnetic properties of materials. Pressure has the effect of altering the magnetic permeability of materials.²⁹ Not only does the saturation magnetic moment change with pressure but the Curie-point can also change. In fact, 30% nickel-iron alloys become nonmagnetic at room temperature under a pressure of 12.5 kilobar.³⁰

As with resistor materials, granular or powdery materials which are polycrystalline will result in magnetic devices which are very susceptible to pressure-induced changes in their electrical properties. Any time there are voids in materials, the the magnetic properties are likely to experience drastic changes with pressure. Purely dimensional changes may also be important in inductive devices.

Core materials for magnetic devices vary greatly in the sensitivity of magnetic parameters, such as permeability, to pressure. Polycrystalline ferrite materials are known to be stress sensitive.³¹ These are used in microwave circuits, in memory devices for computers, and in various logic circuits. If any of these classes of devices are to be operated immersed at high pressure, a careful assessment of their behavior should be made. In addition to the ferrites, powdered iron cores have been found to increase their permeability with pressure, as have molybdenum permalloy dust cores.

The influence of mechanical stress on semiconductors, particularly silicon and germanium, have received much attention in recent years.³⁰ As already mentioned, it is the stress-induced changes in the energy band structure of semiconductors that are reflected in property changes. Both the majority and minority carriers are affected. Silicon and germanium exhibit large piezoresistance effects as compared to metals.³² The majority carrier effects such as piezoresistance result primarily from changes in the relative population of various energy levels due to stress. Phenomenologically, these effects are characterized as changes in effective carrier mobility. As with most crystalline properties, the piezoresistance effects are highly orientation sensitive. Hydrostatic pressure effects on the resistance of semiconductors are, in general, much smaller than shear and uniaxial stress effects. This is due to the fact that hydrostatic pressure does not break up the crystal symmetry.

Minority carriers and their physical properties are stress sensitive. For example, large uniaxial stresses can cause many orders of magnitude increases in the number of minority carries.^{33,34} Minority carrier effects have little influence on the resistance, provided the material is not near its intrinsic resistivity. In 1951, germanium diodes were shown to undergo changes in current-voltage characteristics with hydrostatic pressures. The effects for purely hydrostatic pressures are much lower than for anisotropic stress effects. Purely hydrostatic effects will be negligible for pressure levels of interest in deep ocean applications. It is the cases where the housings induce large nonhydrostatic strains that will be important for the semiconductor devices. Exponential increases in diode or

transistor current are experienced for large uniaxial stresses. Many of the standard semiconductor devices have been studied and their stress dependence characterized.³³⁻³⁷

PHYSICOCHEMICAL EFFECTS ON DEVICES

The immersion of solid-state electronics in a dielectric fluid when there are substantial temperature and pressure variations poses many possible problems. In this section, attention will be focused only on which physicochemical effects of pressure and temperature prevent the successful long-term operation of solid-state devices in a deep ocean environment.

An assessment of the physicochemical effects of pressure and temperature on the following phenomena likely to be encountered should reveal their relative importance.

- Transport of impurities through the passivant.
- Diffusion of contaminants into the passivant.
- Sea-water seepage.
- Impure dielectric fluid.
- Impurities in other materials.
- Rates of diffusion.

The instability of semiconductor devices has long been attributed to the transport of adsorbed ions in the strong electric fields in the regions where p-n junctions intercept the device surface.³⁸ The advent of the planar structure, wherein the sensitive junction regions of the device are passivated with silicon dioxide greatly reduced this problem. The junction is passivated with a material such as silicon dioxide or silicon nitride which protects the device from ionic contaminants. Although the oxide provides a barrier to most ions, it is relatively permeable to alkali ions, particularly sodium.³⁸⁻⁴⁰ The use of resins to provide a moisture seal and hopefully some protection against ionic contamination has been investigated.^{41,42}

Sodium ions have been measured in wet-and dry-grown clean oxides in sufficient amounts to account for oxide instability. Activation analysis of clean thermal oxide films has shown that the sodium ion distribution is 0.5 to 5 ppm (10^{16} to 10^{17} charges/cc), while other investigators using neutron activation analysis have found 1 to 10 ppm of sodium ion.³⁹

To obtain an idea of the order of magnitude of the ionic charge which can cause a drift in devices, consider the threshold voltage in MOS transistors. It can be shown⁴⁰ that the shift in the threshold voltage, ΔV_T , of a MOS structure is given by

$$\Delta V_T = \frac{-Q_{ox}}{C_{ox}} , \quad \text{.....(1)}$$

where Q_{ox} is the charge in the oxide between the gate electrode and the semiconductor surface, expressed as an equivalent surface charge per unit area; and C_{ox} is the capacitance per unit area of the metal gate-semiconductor structure. Rearranging and substituting $C_{ox} = \epsilon_{ox}/d_{ox}$

$$Q_{ox} = \frac{-\epsilon_{ox}}{d_{ox}} \Delta V_T . \quad \text{.....(2)}$$

For a thickness of 0.6μ and a voltage shift of -1 volt

$$Q_{ox} = \frac{-3.35 \times 10^{-11} (-1)}{0.6 \times 10^{-6}} = 5.4 \times 10^{-5} \frac{\text{coul.}}{\text{m}^2} ,$$

or $Q_{ox} = 5.6 \times 10^{14}$ charges/cm². The equivalent of 5.6×10^{14} charges/cc is 3×10^{-2} ppm of sodium ions; i.e.,

$$\frac{5.6 \times 10^{14}}{2 \times 10^{22}} \times 10^6 = 3 \times 10^{-2} \text{ ppm.}$$

Solid-state devices drift within certain tolerances which are acceptable for the present state of the art. It has been established by capacitance-voltage curves and heating under positive and negative bias for MOS devices that device drift is

due to the sodium ion impurities found in technologically clean oxides.^{38,39} It can be concluded that only 1 to 10 ppm of charges, which is the state-of-the-art impurity level, can be tolerated in the oxide layer.

According to Snow, et al,³⁸ the flux, F , of ionic impurities through the oxide layer results from both a concentration gradient, ∇N , and an electric field, E ; i.e.,

$$F = -D\nabla N + \mu EN, \quad \text{.....(3)}$$

where D is the diffusivity (flux per unit concentration gradient), and μ the ionic mobility (flux per unit electric field and unit concentration). In the boundary layer approximation, ion transport at the surface is controlled by diffusion, while ionic drift in the bulk is controlled by the constant electric field. The time rate of impurity concentration at a point just within the oxide surface is expressed adequately by Fick's second law of diffusion,

$$\frac{\partial N}{\partial t} = D\nabla^2 N \quad \text{.....(4)}$$

For the following three boundary conditions, the complimentary error function distribution (erfc) is a solution of Fick's equation:

- The source of ionic impurities is infinite.
- Initially there are no impurities in the oxide layer.
- After a negligible time, the concentration of impurities at the interface, N_0 , remains constant.

The impurity concentration at some point, $N(x)$; expressed as a function of the diffusivity, D ; diffusion time, t ; and the distance from the surface is

$$N(x) = N_0 \operatorname{erfc} \frac{x}{2\sqrt{Dt}} \quad \text{.....(5)}$$

$$\text{or } \log N(x)/N_0 = x/2\sqrt{Dt} \quad .$$

The time required for the accumulation of impurities at some point in the oxide is calculated from the following considerations. The diffusivity is given by

$$D = D_0 \exp (-E_A/kT) \quad , \quad \text{.....(6)}$$

where D_0 is a temperature independent constant and E_A is the activation energy. Based on a simple model,⁴³ D_0 is proportional to: a geometry constant, γ ; the square of the lattice spacing, a^2 , between surface atoms; and a vibrational frequency, ν , the attempt to escape frequency. Using typical numbers for the diffusion of sodium ions through silica; i.e., $\gamma = 1/6$; $a^2 = 2.6 \times 10^{-16} \text{ cm}^2$; $\nu = 10^{13}/\text{sec}$; $E_A = 1$ electron volt, and $kT = 0.33$ electron volt (fluid temperature); $D \approx 10^{-4} \exp (-1/0.03)$, or the diffusivity is of the order of $10^{-16} \text{ cm}^2/\text{sec}$. Oxide layers are of the order of 6×10^{-5} centimeters. The time required for the concentration of impurities to diffuse through the surface (defined as 10% of the oxide layer) and cause a concentration one half that at the interface (between the surface and the liquid) is found graphically. For $N(x)/N_0 = 6 \times 10^{-6}/2\sqrt{10^{-6}t} = 0.5$, t is 3.6×10^5 seconds or 4 days.

Therefore, 4 days are required for the ionic impurities to diffuse through 10% of the width of the oxide layer and cause a concentration one half that at the interface.

While diffusion controls the penetration of ions through the surface, field effects control the drift through the bulk. Snow, et al,³⁸ have shown that for times greater than 4 seconds the concentration at and beyond the boundary layer is negligibly small. This means essentially that, whenever an ion diffuses out of the boundary layer, it is rapidly transported across the rest of the oxide and swept into the vicinity of the oxide-silicon interface.

It can be concluded that a short time is required for the transport of impurities through the surface of the oxide as limited by diffusion, while once the impurities drift beyond the boundary they are rapidly attracted by the electric field to the oxide-silicon interface. Anomalously, high diffusion rates can be expected if channeling through grain boundaries and similar structural defects are present.

Contaminants can be introduced to the devices from a large number of sources. Impurities are attendant in the fluid, electronic hardware, and a host of support materials. As an example of silicone base fluid, VV-D-001078, was analyzed by emission spectroscopy and the test revealed 10 ppm of aluminum, arsenic, boron, calcium, chromium, copper, iron, and magnesium.

The contamination of the fluid through sea-water seepage introduces the following additional ions (see table 8).

Table 8
Composition of Sea Water

Ion	PPM
Borate	710
Bromide	2,380
Calcium	12,000
Chloride	55,200
Carbonate	4,000
Magnesium	37,000
Phosphate	5
Potassium	11,000
Sodium	307,000
Sulphate	77,000

Resin systems for encapsulation can contaminate the fluid since they typically contain 0.01 ppm of silver and beryllium, and 0.1 ppm of boron, bismuth, cadmium, chromium, gallium, germanium, indium, lead, thallium, vanadium, and zinc.⁴⁴ The support material and additional electronic hardware present other sources of contamination.

There are many sources of contaminants in the system and fluid convection will bring these ions in contact with the oxide in a matter of hours. The direction to proceed is to coat the oxide to protect it from moisture and hopefully present a barrier against contaminants. Experiments will be conducted to measure the rate of transport of contaminants through the oxide, and to determine the efflux of contaminants from electronic support hardware. Devices with power on will be immersed in a fluid contaminated with controlled amounts of ions. This will be conducted at various temperatures, but at atmospheric pressure with extrapolation to high pressure. The rates and concentration of ions penetrating the oxide will be analyzed from capacitance-voltage curves. Likewise, electronic support hardware will be immersed in pure fluid and the rates and concentration of contaminants will be analyzed by emission spectroscopy. These tests will be conducted at various temperatures and atmospheric pressure.

The rate of contaminating the passivant at atmospheric pressure can be adjusted for high pressures by considering diffusion theory. As seen previously in the second law of diffusion (equation (4)), the rate of contamination is proportional to the diffusivity which, in turn, is proportional to a geometry factor, lattice spacing, vibrational frequency, and Boltzmann probability (see equation (6)). For a constant geometry factor and Boltzmann probability, the lattice spacing and vibrational frequency would be expected to change slightly with pressure. The rate of device contamination by diffusion should then be slightly different at higher pressures. An assessment of the diffusion of contaminants through the passivant can then be made without the experimental complications of high-pressure apparatus.

DEVICE-HARDENING MATERIALS AND TECHNIQUES

Since true hydrostatic pressure has little effect on most elements of electronic components, the major hardening technique against pressure will be to modify housings and encapsulants to allow the hydrostatic pressure to be transmitted directly to the device elements. In cases where voids either cannot be filled because of the manner in which the device is constructed or because the device operation requires a void (e.g., a vibrating crystal) the component must be potted or hardened mechanically against the pressure. The third type of hardening technique involves the passivation or protection of critical device areas against the fluid environment, be it the fluid or a contaminant therein. Two types of materials are therefore needed: (1) potting

materials which are pressure resistant, and (2) passivation materials which inhibit chemical interaction. Neither of these are without serious problems.

Consider first the case of potting materials for pressure protection. There are several requirements and tradeoffs one must consider. It must be strong enough to withstand the desired pressure, adhere to all types of materials and particularly metal wires and conducting paths, have a reasonable thermal conductivity, have good insulating properties, possess good chemical resistance to both the fluid and the component materials, and have as little shrinkage during curing as possible. The most important practical problems of the above list are the adhesion and shrinkage during curing. The problem of shrinkage during curing can be easily appreciated by considering two simple examples. First, consider the case where it is desired to fill the inside (void) of a housing with a hard material (epoxy, for example). If the epoxy shrinks during curing, it almost certainly will separate from the housing. The very opposite type problem results from potting a component in which shrinkage results in very large internal stresses applied to the device. This problem is well documented in the literature when components were potted.^{41,45-47} It is a very serious problem to say the least. Simple problems often result, such as the breaking off of leads during curing.

Kerlee⁴⁸ categorized potting materials into three types and evaluated them on the basis of adhesion and shrinkage rather than on properties such as humidity resistance, vibration, or heat transfer. A synopsis of types of materials for potting electronics, given in table 9, reveals that a low-density epoxy-bonded syntactic foam adheres best and shrinks the least on curing.

In the case of a passivating or protection coating, the requirements may not be as severe. Chemical inertness is the key for these materials. They must protect against all types of impurities, however, the two most prevalent types are (1) water vapor, and (2) chloride and sodium ions, as well as heavy metals. This type of passivation can be classified as a coating. All types of semiconductor devices utilize some type of passivation scheme. Often a thermal oxide formed during manufacturing operations is used to eliminate ions at critical surfaces and an overcoating of some type of silicone material is used to protect against moisture. In fact, silicone materials are so widely used that they will present a special problem in compatibility with silicone fluids. There are of course the adhesion problems of the passivant with the element; however, a good passivant by definition is a good adherent. It is likely that a good passivant against moisture will also be a good passivant against all other impurities.

Table 9
Synopsis of Potting Materials

Type	Typical Properties
Low-density epoxy-bonded syntactic foam	Low coefficient of thermal expansion, low elastic modulus, high percentage of voids, limited strength
Dense, highly filled, low expansion epoxies	Thermal expansion coefficients closely match component lead materials, strong rigid
Unfilled silicones and urethanes	High thermal expansion coefficient, low elastic modulus

Note: Specific potting materials under consideration include: various types of epoxies, ceramics, and silicones, in addition to glass and diallyl phthalate.

Integrated circuits have passivation and protective layers formed on them during the device fabrication processing steps. These surfaces will in many instances be the first to which additional coatings will be applied. A list of these passivations with typical properties is given in table 10.

TRW, General Electric, and Motorola are evaluating various passivating and encapsulating materials for the Army under contracts DAAB05-70-C-3106, DAAB05-70-C-3109, and DAAB05-70-C-3107. The purpose of these contracts is to develop an inexpensive processing technique meeting the applicable temperature and humidity requirements set forth in MIL-STD-202, method 106. It is our intention to interface with these efforts and evaluate preferred materials likely to provide long-term reliability in the deep ocean environment.

Table 10
Synopsis of Passivation and Protective Layer Materials

Material	Typical Properties
SiO_2	Amorphous layers, relatively permeable to alkali ions, hydrogen and hydroxyl ions Brittle, low yield strength for tension or transverse shear Depending upon method of deposition and thickness, either tensional or compressional stresses may be "built in"
Si_3N_4	Resistant to diffusion of most ions High dielectric constant Chemically inert and mechanically strong Difficult to apply uniformly More difficult to etch than SiO_2 ; makes processing more difficult
Phosphosilicate Glass	Acts as a getter for Na^+ and blocks transmission from outside Improves dielectric strength of SiO_2 Amorphous films
Al_2O_3	Amorphous High dielectric strength Moderate dielectric constant Low surface-state density

SUMMARY AND CONCLUSIONS

The state-of-the-art literature search has been completed. Most of the information given in print on component performance under hydrostatic pressure does not provide sufficient detail for reader-designer. Usually, only general precepts can be extracted from the literature.

The problem of long-term reliability of components operating in electronic equipment where the equipment is flooded by a liquid dielectric has been examined. The various possible interactions,

both physical and chemical, between components, dielectric fluid and electronic materials, housing, and sea-water and acoustic environments were examined and tentative estimates made as to their importance.

The possibility of constructing a wet-electronics package having positive or neutral buoyancy was investigated and the conclusion reached that it probably can be done.

A scheme for presenting information on materials, devices, configurations, functions, failure modes, etc, which will be used to analyze and present data relative to pressure and fluid effects has been devised and will be used as an aid in choosing the coatings and potting materials that will be used for experiment.

The conclusion has been reached that, in principle, the pressure- or stress-type problems relating to the devices can be solved by potting or coating. If the coatings and pottings also have satisfactory chemical properties, it is expected that a technology can be developed which will permit the reliable operation of electronic equipments when immersed in a dielectric fluid at high hydrostatic pressure.

PLANS FOR FUTURE WORK

- A semiconductor device sensitive to ionic contamination will be chosen as a detector. Experiments will be carried out to calibrate the detector in terms of various contaminant concentrations in the dielectric fluid and operational time.
- The calibrated detector will be utilized to establish accelerated testing procedures to define experimentation with coatings and pottings.
- Fluid-coating compatibility experiments will be performed.
- Fluid-potting compatibility will be performed.
- Preferred device types, in terms of manufacturer's coating, bonding technique, and geometry will be established.
- Reliability criteria will be developed.
- A test vehicle will be developed.

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